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L1: Entry 2 of 2

File: DWPI

Jul 8, 1997

DERWENT-ACC-NO: 1997-397335

DERWENT-WEEK: 200203

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TITLE: Material for filling living body - comprises titanium (alloy) intermediate metal carbide layer and diamond layer

## PATENT-ASSIGNEE:

ASSIGNEE	CODE
KYOCERA CORP	KYOC

PRIORITY-DATA: 1995JP-0340117 (December 27, 1995)

## PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
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<input type="checkbox"/> JP 3236768 B2	December 10, 2001		005	A61L027/00

## APPLICATION-DATA:

PUB-NO	APPL-DATE	APPL-NO	DESCRIPTOR
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JP 3236768B2	December 27, 1995	1995JP-0340117	
JP 3236768B2		JP 9173437	Previous Publ.

INT-CL (IPC): A61 F 2/30; A61 L 27/00

ABSTRACTED-PUB-NO: JP 09173437A

## BASIC-ABSTRACT:

Material for filling living body comprises titanium or titanium alloy, intermediate layer comprising metal carbide non-toxic to living body and a diamond layer.

ADVANTAGE -High adhesion strength can be obtained.

CHOSEN-DRAWING: Dwg. 1/7

TITLE-TERMS: MATERIAL FILL LIVE BODY COMPRISE TITANIUM ALLOY INTERMEDIATE METAL CARBIDE LAYER DIAMOND LAYER

DERWENT-CLASS: D22 P32 P34

CPI-CODES: D09-C;

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L1: Entry 1 of 2

File: JPAB

Jul 8, 1997

PUB-NO: JP409173437A  
DOCUMENT-IDENTIFIER: JP 09173437 A  
TITLE: BIOPROSTHETIC MEMBER

PUBN-DATE: July 8, 1997

## INVENTOR-INFORMATION:

NAME	COUNTRY
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ICHINOMIYA, MASARU	

INT-CL (IPC): A61 L 27/00; A61 F 2/30

## ABSTRACT:

PROBLEM TO BE SOLVED: To improve adhesion strength of a diamond layer by applying the diamond layer on the surface of a base body comprising titanium or a titanium alloy through an intermediate layer comprising diamond and metal carbide.

SOLUTION: An intermediate layer 2 (silicon carbide) comprising at least diamond 4 and a metal carbide 5 which has biological compatibility is provided between a diamond layer 3 and a base body 1. Metal carbide 5 included in the intermediate layer 2 is solid solution-coupled with titanium in the base body 1 at a boundary surface between the intermediate layer 2 and the base body 1. In the meanwhile, covalent binding occurs between carbons in diamond included in the intermediate layer 2 and diamond in the diamond layer 3 at a boundary surface between the intermediate layer 2 and the diamond layer 3. In addition, covalent binding occurs between carbons composing diamond and metal carbide in the intermediate layer 3. Because covalent binding has stronger binding force than ion binding, adhesion strength of the diamond layer can be improved.

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試料 No.	基体組成 金属元素の前の數 字は含有量(wt%)	成膜 条件 注)	中間層の 成分	厚み 中間層	厚み 表面層	臨界荷重 (kgf)	ラマン 分光分析 $I_2/I_1$	表面粗さ (μm)	ピン摩耗量 (g)	摩擦係数
1	90Ti-6Al-4V	表1	SiC, TiC	1.2	4.8	110	0.43	0.4	0.008	0.10
2	90Ti-8Al-1V-1Mo	表1	SiC, TiC	1.1	4.8	110	0.20	0.5	0.010	0.11
3	85Ti-10V-2Zr-3Al	表1	SiC, TiC	1.1	4.9	110	0.52	0.3	0.008	0.09
4	100Ti	表1	SiC, TiC	1.2	4.8	110	0.42	0.4	0.011	0.10
5	77Ti-15Mo-5Zr-3Al	表1	SiC, TiC	1.2	4.9	110	0.40	0.4	0.010	0.11
* 6	90Ti-6Al-4V	表1'	なし	—	5.8	5	0.30	0.5	—	0.11
* 7	90Ti-8Al-1V-1Mo	表1'	なし	—	5.7	5	0.35	0.4	—	0.10
* 8	77Ti-15Mo-5Zr-3Al	表1'	なし	—	5.7	5	0.36	0.4	—	0.10

\*印は本発明の範囲外の試料を示す。

注) 表1'は、表1からSL(CH<sub>4</sub>)<sub>x</sub>ガスを除いた条件である。

【0029】また、これらの部材に対して、耐摩耗摺動特性（ピンの摩耗量及び摩擦係数）をピンオンディスク法により評価した。摺動試験の条件は、室温、大気中、無潤滑において荷重39.2N、摺動速度2m/sで、24時間で行った。ピンはアルミニウム製のものを用いた。試験前後のアルミニウムピンの重量変化でピンの摩耗量を評価した。ピンの摩耗量と摩擦係数を表2に示す。

【0030】さらに、ビッカース硬度計を用いて膜に荷重をかけて基体表面から膜を浮かせ、膜と基体との付着力を評価した。膜に剥離が生じはじめた荷重（臨界荷重）を測定した結果を表2に示す。

### 【0031】

【発明の効果】叙上のように、本発明の生体補綴部材は、ダイヤモンドと金属炭化物とからなる中間層を介してダイヤモンド層を形成したことにより、中間層とダイヤモンド層を構成する炭素が共有結合し、他方、中間層と基体を構成する金属材料が固溶固着するんで、密着強度が大きく、上記ダイヤモンド層が剥離しにくく、さらにダイヤモンド層が微結晶ダイヤモンドを含むことにより、耐摩耗性、滑動性に優れ、したがって、生体に安全で長期の使用に耐えうるという優れた効果を奏するものである。

### 【図面の簡単な説明】

\* 【図1】本発明の生体補綴部材の構造を説明するための模式図である。

20 【図2】本発明実施形態としての大腿骨部材の斜視図である。

【図3】本発明実施形態としての骨頭ボールの側面図である。

【図4】本発明実施形態としての人工歯根の側面図である。

【図5】大腿骨用リーマの側面図である。

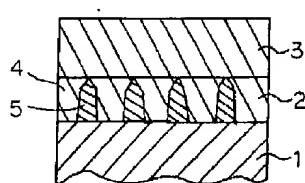
【図6】歯科用リーマの側面図である。

【図7】ラマン分光分析の結果を示すグラフである。

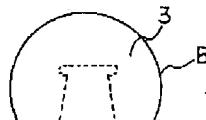
### 【符号の説明】

30	1	基体
	2	中間層
	3	ダイヤモンド層
	4	ダイヤモンド
	5	金属炭化物
	F	大腿骨部材
	f	摺動面
	B	骨頭ボール
	R	人工歯根
	r	歯肉当接部
40	T	大腿骨用リーマ
*	C	人工歯根

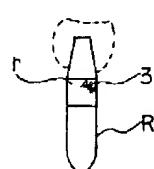
【図1】



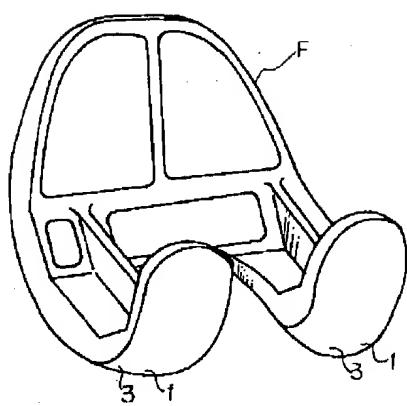
【図3】



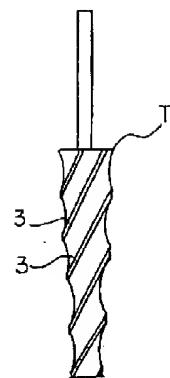
【図4】



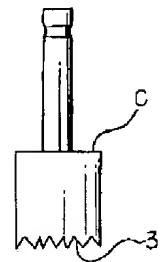
【図2】



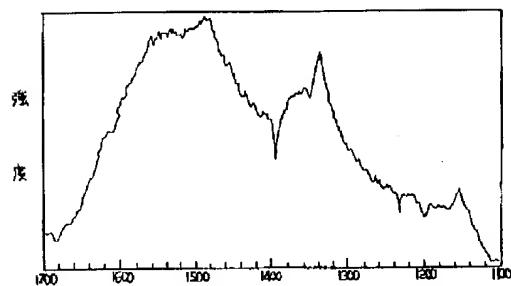
【図5】



【図6】



【図7】



**Patent/ public disclosure document**

[Abstract(made by the applicant)] [Claims] [Detail Description] [Drawing Description]  
PATOLIS will not assume the accuracy or the reliability of the translation

JP 9-173437

(57)

## [ABSTRACT]

## [PROBLEM TO BE SOLVED]

As against a living body prosthetic member to become than titanium or titanium alloy, it is had with big coherence strength, and the diamond layer which can be superior to strength, \*dosei is formed.

## [SOLUTION]

In the surface of substrates to become than titanium or titanium alloy, the middle class that it was from a diamond and metal carbide was gone through, and a diamond was adhered by.

---

## [WHAT IS CLAIMED IS]

## [Claim 1]

In the surface of substrates to become than titanium or titanium alloy, the middle class that it is from a diamond and metal carbide without *seitaiigaisei* is gone through, and it is the living body prosthetic member that a diamond is adhered by, and it is.

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## [DETAILED DESCRIPTION OF THE INVENTION]

## [0001]

## [TECHNICAL FIELD OF THE INVENTION]

The present invention relates to the living body prosthetic member which a function substitutes for fall or one part of a bone and a joint of the person that it is lost, and sharp pain increased by a disease of transformation-related joint symptoms.

## [0002]

## [PRIOR ART]

As for the diamond, most liquid, gas have corrosion resistance, the hardness is high, and a compatibility to the living body is big, and it wants to be done, and use as living body materials is the materials which have been expected.

## [0003]

At Japanese Patent Publication No. 3-64142 bulletin, invention to relate to the living body business member which jacketed a substratum comprising metal materials in letter of direct diamond and diamond carbon with the thing which used a diamond or letter of diamond carbon as living body materials is described.

## [0004]

## [a prior-art problem]

However, There were the following problems in the prior art. In other words, Membrane in itself is what direct, formed a coating membrane in a substratum by various CVD method such as tungsten filament CVD method and rF plasma CVD method although high hardness, a characteristic such as for example a low coefficient of friction are provided, and, by the prior art, a material and coherence strength with a membrane that it is in a substratum are lacked, when coherence strength of a substratum and canning layer was small, and it was going to be used to a slide portion particularly, there is danger that a coating membrane exfoliates, it is the fact that do not reach practical use.

## [0005]

For the reason why these substrates and coherence strength with a coating membrane are low, that getting wet sex with other materials is bad is mainly given to the rigid carbon which seem to be letter of diamond and diamond carbon.

## [0006]

## [OBJECT OF THE INVENTION]

In view of the prior-art problem, the present invention is had with big coherence strength for a substratum to become than titanium or titanium alloy and is directed to that the living body prosthetic member which formed the diamond layer which can be superior to strength, \*dosei is provided.

[0007]

#### [MEANS TO SOLVE THE PROBLEM]

A living body prosthetic member of the present invention went through the middle class that it was from a diamond and metal carbide in the surface of a substratum to become than titanium or titanium alloy to solve the prior-art problem, and diamond layer was adhered by.

[0008]

#### [OPERATION]

With the thing which a living body prosthetic member of the present invention goes through the middle class that it is from a diamond and metal carbide without *seitaigaisei* in the surface of substrates to become than titanium or titanium alloy, and formed diamond layer, which materials do not have *seitaigaisei* either and, as for the diamond, sliding sex with sliding materials such as polyethylene is good. Even more particularly, Metal carbide and titanium of substrates included in a middle class in an interface of a middle class and substrates do *ko\*ketsugo*, a diamond and a diamond of diamond layer included in a middle class in an interface of the other, a middle class and diamond layer do carbon *doushiga* covalent bond, even if, even more particularly, it is put in a middle class, carbon *doushiga* covalent bond to compose a diamond and metal carbide is done. By this, Coherence strength with diamond layer and substrates improves.

[0009]

Thus, Detachment of materials and outbreak of abrasion powder are restrained partly, it is by security and a big living body prosthetic member of the durability in the living body very.

[0010]

#### [MODE FOR CARRYING OUT THE INVENTION]

As follows, An embodiment of the present invention is explained by means of a figure. A living body prosthetic member of the present invention comprises substrates 1 and middle class 2 and diamond layer 3 as shown in FIG. 1, even if diamond layer 3 is a membrane covering up entire surface of substrates 1 or one part of surface option may be covered. As for substrates 1, a living body compatibility is bought along with high intensity, *koutubosei* by the use environment. Thus, According to the current invention, it is important that titanium or titanium alloy is used as the materials which are with a high intensity and *koutubosei*.

[0011]

Even more particularly, According to the current invention, diamond layer 3 is formed on the sliding surface in a living body prosthetic member as slide member, and coherence strength to be enough so that detachment is not generated should be got, it is important to establish middle class 2 that, at a minimum, it is from diamond 4 and metal carbide 5 having a living body compatibility between diamond layer 3 and substratum 1 as shown in FIG. 1.

[0012]

The reason that coherence strength with substrates 1 improves is regarded as diamond layer 3 as follows by the formation of such middle class 2.

[0013]

Metal carbide and titanium of substrates 1 included in middle class 2 in an interface of middle class 2 and substrates 1 do metal how, and *ko\*ketsugo* is done, a diamond and a diamond of diamond layer 3 included in middle class 2 in an interface of the other, middle class 2 and diamond layer 3 do carbon *doushiga* covalent bond , even more particularly, carbon *doushiga* covalent bond to compose a diamond and metal carbide in middle class 2 is done. If it says as concerns embodiment of combination, covalent bond has strong combination than an ionic complex. Strong combination power is provided in a diamond comprising covalent bond of carbon. For a carbon compound without *seitaigaisei*, there are silundum and carbonization titanium , of these, silundum is covalent bond-related carbide.

[0014]

In addition, Because diamond 4 is distributed in the shape of an island, as shown in FIG. 1 rather than what diamond 4 in this middle class 2 and metal carbide 5 separate in the shape of the stratum, and there is,

improvement of coherence strength by an anchor effect can be expected. In addition, As for middle class 2, it is desirable for it to be formed with thickness of 0.5-5 mu in particular. In addition, As for diamond layer 3 and total thickness 1-100 with middle class 2, 2-20 mu is particularly desirable mu.

[0015]

In addition, Surface coarseness of diamond layer 3, of a membrane, it is crystalline, and it is affected greatly. Therefore, Because existence succeeds in getting slight polycrystalline diamond, surface coarseness of a membrane can be controlled small. Besides, Slight polycrystalline diamond is good to an abrasion resistance from an abrasion resistant point.

[0016]

Such a slight polycrystalline diamond can ensure existence in Raman light spectrum analysis for a peak of  $1160 \text{ cm}^{-1}$ . Thus, There are a lot of slight polycrystalline diamond so that this peak is big, surface coarseness of a membrane tends to shrink.

[0017]

Next, For a method to make a living body prosthetic member of the present invention, hydrogen and carbon component gas and carbon component gas are introduced as a source gas in an installed reaction chamber of substrates in vapor phase growth method, diamond and metal carbide, middle class 2 that, for example, it is from silundum can be formed by what is activated, if, even more particularly, supply of silicon component gas is stopped, diamond layer 3 can be formed.

[0018]

As follows, Application in the present invention is shown in FIG. 2-FIG. 6. FIG. 2 shows femoral component F of an artificial knee joint, this femoral component F goes through two middle class (not shown) in sliding aspect f composing a joint side, and diamond layer 3 is jacketed , for example, polyethylene and ceramic, sliding sex for metal materials are raised.

[0019]

FIG. 3 shows grit ball B composing an artificial hip joint, this grit ball B goes through two middle class (not shown) to an outer surface, and diamond layer 3 is jacketed, sliding sex was raised.

[0020]

FIG. 4 shows artificial root R, this artificial root R went through two middle class (not shown) in gingiva abutment r, and diamond layer 3 was jacketed. By this, Elution of metal ions is suppressed, darkening prevented a situation to develop to a gingiva, and an abrasion resistance as opposed to a brush was improved.

[0021]

As for figure 5 and figure 6, FIG. 5 shows dentistry business reamer C in thighbone use reamer T, FIG. 6 as application to an edged tool for this technical operations respectively. Two middle class (not shown) is gone through on a blade part respectively, and diamond layer 3 is jacketed. By this, It can make the sharpness of a blade last for a long term.

[0022]

In addition, In an application shown in FIG. 2-FIG. 6, substrates are titanium, titanium alloy both.

[0023]

#### [EXAMPLE]

A source gas was introduced in a reactor, and pressure force in a reaction chamber was set to 0.1torr, substrates temperature 800 degrees Celsius. A kind of a source gas, flow quantity are shown for table 1. It makes apply a magnetic field of strength of greatest 2k gauss by ECR plasma CVD method, under conditions of microwave output 3.0KW, Ti -6% Al-4 % V alloy ((specimen No 1) that the ratio of an alloy layered all on weight %) substrates as follows.) It is shown in table 1 along with progress of time layering source gas flow quantity in layering and a change of pressure force. In addition, A Raman light analysis result was shown in FIG. 7 about specimen No 1.

[0024]

[TABLE 1]

経過時間 (hr)	0 → 10	10 → 50
CH <sub>4</sub> (SCCM)	2	2
CO <sub>2</sub> (SCCM)	4	4
Si(CH <sub>3</sub> ) <sub>4</sub> (SCCM)	0.5	0
H <sub>2</sub> (SCCM)	194	194
圧力 (Torr)	0.1	0.1

[0025]

In addition, Substrates were exchanged with Ti -8% Al-1 % MO alloy, Ti -10% V-2 % Fe -3% Al alloy, Ti 100%, Ti -6% Al-4 % V alloy, and coating was done by similar technique (No 2,3,4,5). All did an abrasion process for 20 as against made specimen.

[0026]

Even more particularly, The others which did not supply Si (CH<sub>3</sub>)<sub>4</sub> among source gas composition shown in table 1 as a comparative example made comparison specimen (No 6,7,8) by technique same as the said article.

[0027]

A search aspect was accompanied by by the X-ray diffraction measurement, and, as against a provided membrane, a result was shown for table 3. In addition, The spectrum measurement was done by Akira slight Raman minute light method, and peak strength I<sub>1</sub> of 1333 ? 10cm<sup>-1</sup>s, the strength ratio (I<sub>1</sub>/I<sub>2</sub>) with peak strength I<sub>2</sub> of 1160 ? cm<sup>-1</sup>s were shown for table 2. Even more particularly, Surface coarseness (Rmax) was measured with syoku needle-style surface coarseness meter.

[0028]

[TABLE 2]

試料 No	基体組成 金属元素の前の數 字は含有量(wt%)	成膜 条件 (注)	中間層の 成分	厚み 中間層 /外被 層 (μm)	臨界荷重 (kg重)	ラマン 分光分析 I <sub>2</sub> /I <sub>1</sub>	表面粗さ (μm)	ピン摩耗量 (g)	摩耗係数	
1	90Ti-6Al-4V	表1	Si <sub>3</sub> N <sub>4</sub> , SiC, TiC	1.2	4.8	110	0.43	0.4	0.008	0.10
2	90Ti-8Al-1V-1Mo	表1	Si <sub>3</sub> N <sub>4</sub> , SiC, TiC	1.1	4.8	110	0.20	0.5	0.010	0.11
3	85Ti-10V-2Fe-3Al	表1	Si <sub>3</sub> N <sub>4</sub> , SiC, TiC	1.1	4.9	110	0.52	0.3	0.008	0.09
4	100Ti	表1	Si <sub>3</sub> N <sub>4</sub> , SiC, TiC	1.2	4.8	110	0.42	0.4	0.011	0.10
5	77Ti-15Mo-5Zr-3Al	表1	Si <sub>3</sub> N <sub>4</sub> , SiC, TiC	1.2	4.9	110	0.40	0.4	0.010	0.11
* 6	90Ti-6Al-4V	表1*	なし	—	5.8	5	0.30	0.5	—	0.11
* 7	90Ti-6Al-1V-1Mo	表1*	なし	—	5.7	5	0.35	0.4	—	0.10
* 8	77Ti-15Mo-5Zr-3Al	表1*	なし	—	5.7	5	0.36	0.4	—	0.10

\*印は本発明の範囲外の試料を示す。

注) 表1\*は、表1からSi(CH<sub>3</sub>)<sub>4</sub>ガスを除いた条件である。

[0029]

In addition, As against these member, an abrasion sliding characteristic (quantity of abrasion and an abrasion coefficient of a pin) resistance was evaluated by a pin on disk method. The condition of a sliding examination went in room temperature, no lubrication out of the atmosphere in load 39.2N, sliding speed 2m/sec, 24 hours. The pin used a thing made by aluminum. Quantity of abrasion of a pin was evaluated by a weight change of the aluminum pin that an examination was forward and backward. Quantity of abrasion and a coefficient of friction of a pin are shown for table 2.

[0030]

Even more particularly, Load was sprinkled on a membrane by means of a *bikkasu* sclerometer, and saved a membrane from a substrate surface, and a membrane and adhesive power with a substratum were evaluated. The result that measured load (critical load) that detachment has begun to produce on a membrane is shown for table 2.

[0031]

#### [EFFECT OF THE INVENTION]

Like the above mentioned, the carbon which composes a middle class and diamond layer by a middle class to become from metal carbide intervenes between a diamond and a living body prosthetic member of the present invention, and having formed diamond layer does covalent bond, *ko\*kochaku surunnode*, coherence strength are big, and metal materials composing the other, a middle class and a substratum are hard to exfoliate the diamond layer , even more particularly, diamond layer be superior to abrasion resistance, *\*dosei* by a thing including slight polycrystalline diamond, and it wants to be done, and it is safe, and a superior effect to be able to bear long-term use is played in the living body.

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#### [BRIEF DESCRIPTION OF DRAWINGS]

[FIG. 1]

It is a schematic block diagram to explain structure of a living body prosthetic member of the present invention.

[FIG. 2]

It is a perspective diagram of femoral component as a present invention embodiment.

[FIG. 3]

It is a side elevation of a grit ball as a present invention embodiment.

[FIG. 4]

It is a side elevation of an artificial root as a present invention embodiment.

[FIG. 5]

It is a side elevation of a reamer for thighbones.

[FIG. 6]

It is a side elevation of a reamer for dentistry.

[FIG. 7]

It is a graph showing a result of Raman light analysis.

#### [DENOTATION OF REFERENCE NUMERALS]

A reamer C artificial root for one two three four five substrates middle class diamond layer diamond metal carbide F femoral component f sliding aspect B grit ball R artificial root r gingiva abutment T thighbones

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